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January 31, 1973

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Dr. Julie C. Yang
Senior Group Leader
W. R. Grace & Co.
Rock Processing Chemicals
Construction Products Division
Cambridge, Massachusetts 02140

Dear Julie:

Per your letter of January 11, 1973, and subsequent telephone conversations with Dr. Arnold Rosenberg and you, I am enclosing a report providing an analysis of asbestos (tremolite and actinolite) content of seven (7) unknown samples as well as an operating procedure for determining asbestos content in Monokote samples. The method employed has demonstrated a 2σ confidence minimum detectable limit of 0.15 weight percent, which I think is especially good for a procedure based upon X-ray diffraction methods.

The overall cost for this work, which we will bill to your P. O. No. 41574, is \$1800. This includes the small carryover from the previous task (December 19, 1972), diffraction scans of 12 samples, Method B and C point count data for seven (7) samples, Method C point count data for an additional seven (7) samples, and finally, specification of a measurement procedure.

I have retained all submitted samples and prepared X-ray samples should they be required in the future.

Very truly yours,

Ed

Edward T. Peters

/mc

Enclosure

CAMBRIDGE, MASSACHUSETTS

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January 30, 1973

PROCEDURE FOR MEASURING ASBESTOS CONTENT OF
MONOKOTE MIXTURES FOR W. R. GRACE & CO.

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SUMMARY

An X-ray diffraction procedure has been developed for determining the presence of tremolite and actinolite forms of asbestos in commercial mixtures of vermiculite and gypsum, such as monokote. Based upon the results from known chemistry standards, asbestos can be identified in these products with a minimum detectable limit of 0.12 weight percent for tremolite and 0.15 weight percent actinolite. Of the seven samples submitted for measurement of asbestos content, all were found to have less than the minimum detectable limits of asbestos, with the following exceptions: African #3 - 1.90% tremolite and Kearney #3 - 0.30% tremolite.

INTRODUCTION

In December 1972, members of the Construction Products Division, W. R. Grace, Inc., reviewed with us a need for accurately determining the asbestos content of various commercial product mixtures, such as monokote. It was agreed that X-ray diffraction analysis appeared most practical. Analysis of several standards (0.5, 1.0 and 2.0% tremolite in monokote) revealed that the presence of asbestos could be detected by a diffraction scan strip chart recording. To explore the possibility of improving the sensitivity of the X-ray method, Arthur D. Little, Inc., conducted a second set of experiments based upon the fixed count X-ray method. This proved successful, providing a minimum detectability limit of 0.12 weight percent tremolite in monokote. These results were presented in our report dated December 19, 1972.

On January 11, we were asked to:

- 1) Prepare a calibration curve for the quantitative analysis of actinolite, utilizing
 - a) Fixed count procedures, as before.
 - b) Area under the curve, after slow scans.
- 2) Conduct an analysis of several expanded vermiculite samples and of the monokote product prepared at various locations from Libby ore to determine tremolite and actinolite content.

In our preliminary work, it became clear that the actinolite standard mixes were different than the other samples, in that they resisted dispersion in mixing with amyl acetate. Subsequently, it was determined that the standards were improperly prepared and a new set was submitted. On January 26, 1973, a final expanded vermiculite sample was submitted for analysis. This did not have the same pre-treatment as the other samples, resulting in a much coarser particle aggregate size.

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Arthur D Little Inc

Samples were prepared and analyzed similar to the earlier work. A description of the methods used and procedural outline are presented.

EXPERIMENTAL PROCEDURE

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It was assumed that the pre-treatment provided by W. R. Grace resulted in uniform, well blended samples. X-ray samples were prepared by mixing 20-50 mg of the powder mixture with amyl acetate to make a slurry. Thorough mixing was carried out in a mortar and pestle after which the slurry was poured onto a glass microscope slide and dried. All X-ray diffraction data were carried out with a copper X-ray tube operated at 40 kV and 20 ma. The apparatus utilized a post beam monochromator equipped with a graphite crystal to minimize scattered, background radiation. Based upon the previous study and upon information from diffraction scans of the monokote mixtures and pure asbestos standards, it was determined that the most suitable diffraction line positions for the asbestos peaks free from interference from other peaks were at:

Tremolite - $2\theta = 28.5^\circ$

Actinolite - $2\theta = 12.4^\circ$ and 28.5°

X-ray data were collected according to three basic procedures, as follows:

A. Diffractometer Scan

Scanning was conducted at a rate of $1^\circ/\text{minute}$ over the range of $2\theta = 4-50^\circ$. This scan exhibited all diffraction peaks. As shown earlier, this approach permitted detecting the presence of 1% tremolite (at $2\theta = 28.5^\circ$), and from the present work, 1/2% actinolite (at $2\theta = 28.5^\circ$). As both peaks occur at 28.5, the direct scan approach can only say one or the other or both forms of asbestos are present in monokote in excess of 1%.

B. Area Display (Slow Scan)

Scans were made at $1/4^\circ/\text{minute}$ over the range of interest ($2\theta = 11.9-12.9$ and $28.0-29.0^\circ$). As there was little "area under the curve" for most samples, equivalent data were collected by measuring counts in 60 seconds at 28.0 and 29.0° as background and counts in a 120 second scan over the peak from 28.0° to 29.0° as peak. The signal is then taken as peak minus background.

C. Point Count

Data were collected at fixed positions from peak (12.4 and 28.5) and background (11.9 and 28.0), recording the time (seconds) to collect 6400 counts, providing a 2 σ probable error of 1.68%. In the case of the expanded vermiculite samples, the 28.5° area of interest was influenced by the tail of an adjacent, broad peak; for these samples, background was taken to be the average of measurements at 28.0 and 29.0°.

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EXPERIMENTAL RESULTS

Diffraction scans for the 12 submitted samples are attached. Examination of the traces showed the expected peaks in all cases. The three expanded vermiculite samples all showed variation from one another, which is attributed to small differences in the composition of the ore or processing variables. The scans of monokote prepared at four locations were essentially identical. The results from the various methods of analysis are given below, with measured data presented in Table 1:

Method A

From the standards, a peak at 28.5° is observed with as little as 0.5% actinolite or (from the previous work) 1.0% tremolite. However, at least 2.0% actinolite is required to observe the peak at 12.4° . Based upon the higher backgrounds and interfering tails of adjacent peaks present in the monokote and expanded vermiculite samples, it is concluded that diffraction scans are suitable for identifying the presence of asbestos in quantities of 2 weight percent or greater.

Method B

As can be inferred from the data presented in Table 1, slow scanning fails to exhibit a peak distinguishable from background. Using the more exacting measurement of counts collected at background (120 sec at 11.9° and 120 sec at 12.9° or 120 sec at 28.0° and 120 sec at 29.0°) and from background plus peak (240 sec for scan from $11.9 \rightarrow 12.9^\circ$ and $28.0 \rightarrow 29.0^\circ$), one observes in Table 1 that background is generally higher than peak count. Although no clear explanation can be provided for this, it is assumed that background is not uniform over the range scanned. The fact that peak signal is so low, precluding a measurable area above background, rules out this approach for determining asbestos content in monokote samples.

Method C

Experimental data collected according to the Method C procedure are presented in Table 1. Each measurement is converted to a counts/second basis, with appropriate correction for the difference in background counting rate at P and B positions as determined from the monokote blank. A plot of signal (i.e., peak less background) versus composition for the various standards is presented in Figure 1. With the exception of one datum point, the tremolite data is in excellent agreement with the earlier calibration curve (December 19 report), giving considerable credence to the experimental approach that has been employed.

The actinolite data show some scatter. The curve at 12.4° (with a 2σ - confidence, minimum detectable limit of 0.15%) is employed to identify the presence of actinolite. From Figure 1, the corresponding count rate for the 28.5° actinolite peak is determined and subtracted from the corrected 28.5° signal. Any remaining signal is attributed to tremolite.

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Experimental data for the seven unknown samples is also presented in Table 1. As the 28.5° peak position occurred on the tail of a major expanded vermiculite peak, a more appropriate background was obtained by averaging data collected at 28.0 and 29.0° positions. Even this approach resulted in an over-correction for background. This high background difficulty is the result of a broad expanded vermiculite peak being present at 26.7° in the North Carolina ore employed for standards, where there was no interference from peak tails at 28.0°, whereas it is present at 27.3° in the Libby ore resulting in a peak tail at 28.0°. As a consequence, the absence of a peak at 28.5° in the four monokote samples using Libby ore was inferred by a measured peak to background ratio of 0.86 for all four samples.

The three expanded vermiculite samples showed no signal at 12.4°, precluding the presence of actinolite. A measured signal at 28.5° was therefore attributed to tremolite corresponding to 1.95% and 0.30% for the African #3 and Kearney #3 samples, respectively.

RECOMMENDED PROCEDURE

Based upon the experimental results described above, the following procedure is recommended for determining the presence of asbestos (actinolite and/or tremolite) in monokote samples:

1. Mix 20 to 50 mg monokote mix with 20 to 30 drops amyl acetate, mix in mortar and pestle, pour onto a glass slide (covering an area of 4-6 cm²), and allow to dry.
2. Employing a Philips vertical diffractometer equipped with a copper target X-ray tube operated at 40 kV and 20 ma, 1° divergence slit, 0.001 inch receiving slit and graphite-crystal post beam monochromator,* collect the following data:
 - a. Measure time to collect 6400 counts at $2\theta = 12.4^\circ$ and convert to counts/second = P1
 - b. Measure time to collect 6400 counts at $2\theta = 11.9^\circ$ and convert to counts/second = B1
 - c. Calculate $S1 = P1 - B1$
 1. If $S1 = 0.4$ or lower, assume no actinolite is present.
 2. If $S1 > 0.4$, read % actinolite from curve (1), Figure 1. Also, read counts/second at same % actinolite from curve (2) and call S2.*
 - d. Measure time to collect 6400 counts at 28.0, 28.2, 28.5, 28.8 and 29.0°; convert to counts per second; plot counts/second versus 2θ ; draw smooth curve through points at $2\theta = 28.0, 28.2, 28.8$ and 29.0°; take difference between 28.5° point and the smooth curve and call P2.

*Other experimental apparatus could of course be used, but would probably require new calibration curves.

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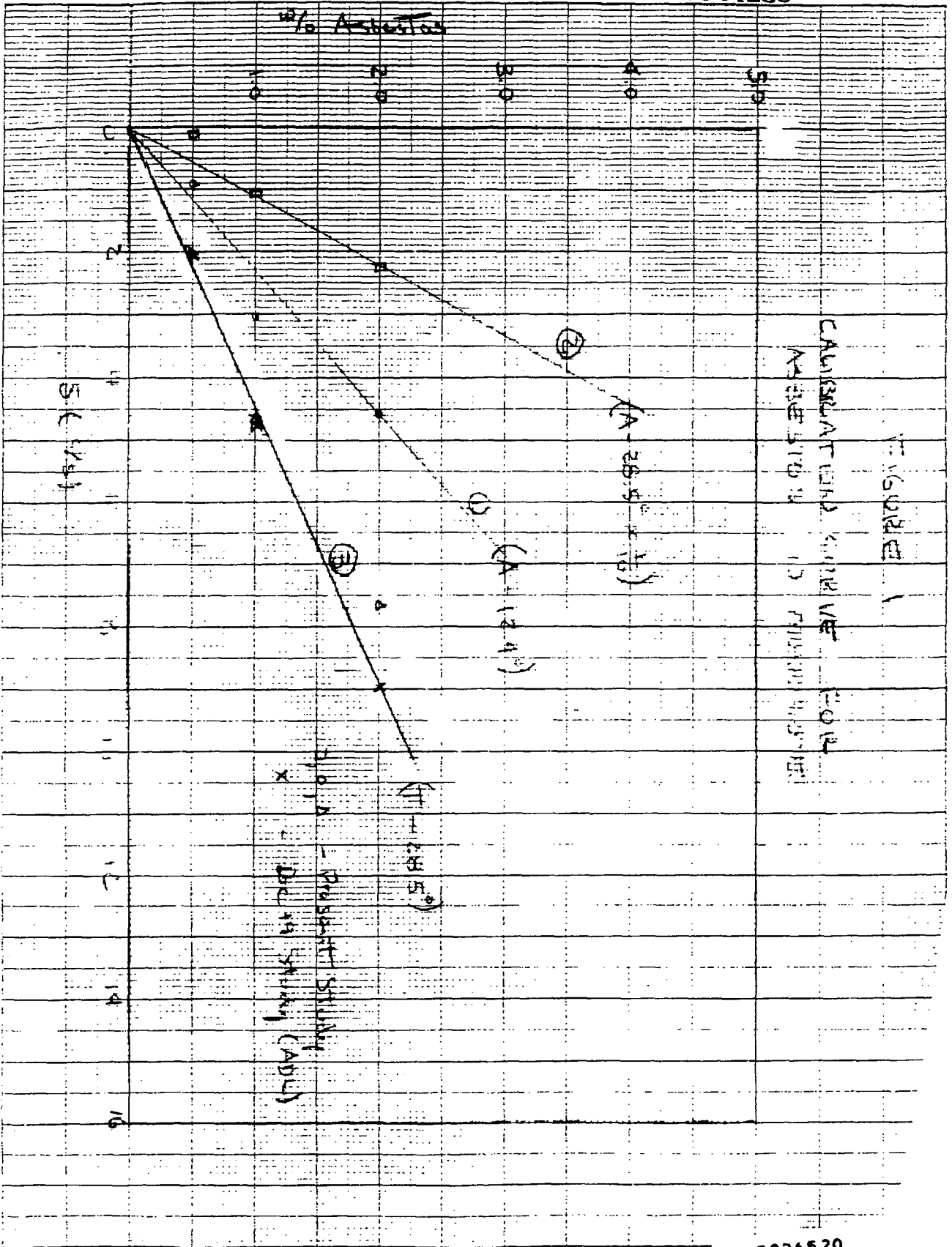
- e. Calculate $S_2 = P_2 - S_2^*$
1. If $S_2 = 0.7$ or less, assume no tremolite is present.
 2. If $S_2 > 0.7$, read % tremolite from curve (3), Figure 1.
3. For procedure as presented above, the minimum detectable limit of tremolite is 0.12 and actinolite is 0.15 weight percent, respectively.

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